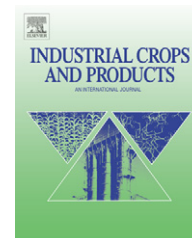


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# Oil extraction from lesquerella seeds by dry extrusion and expelling

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## ABSTRACT

Whole lesquerella seeds with 6% (as is) and 12% moisture content (MC) were extruded at different residence times by varying screw speeds and feed rates. The temperature of the extrudate was recorded and its MC was determined. The extent of seed cooking was evaluated by measuring the protein solubility and thioglucosidase (TGSase) activity in the extrudate. Uncooked whole seeds (UWS), whole seeds cooked in seed cooker (CWS), and extrusion-cooked seeds (ECS) were screw pressed and the crude oils were analyzed for foats, free fatty acid (FFA), phosphorus, calcium, magnesium, and sulfur. The screw speed and feed rates employed resulted in residence times ranging from 22 to 110 s. The corresponding exit temperatures of the extrudates ranged from 88 to 143 °C. Seeds with 6% initial MC dried to 4.3% at extrudate temperatures  $\leq 125$  °C regardless of residence time, while seeds with 12% initial MC came out at 7–9% MC. Extruding seeds with 6 and 12% starting MC for 34 and 41 s, respectively, provided the same degree of cooking as that of 12% MC CWS. All CWS and ECS tested negative for TGSase activity. ECS with 6% initial MC generated much higher foats (6.4–9.4%) in the oil compared with that of the 12% MC ECS (1–1.7%). The crude oils from CWS had the lowest FFA content at 1.25%. Crude oils from UWS and ECS had FFA ranging from 1.4–2.8%. The crude oil from 12% MC CWS had 374 ppm sulfur which was 3–8 $\times$  higher than what were found in crude oils from 6% MC CWS and ECS. The highest P (23 ppm), Ca (14 ppm), and Mg (6 ppm) levels in the crude oil were from 12% MC CWS, which were comparable to total degummed oils. An 81% oil recovery from 6% MC ECS (22 s residence time) was obtained at 19 rpm expeller screw speed. Increasing the expeller's screw speed from 19 to 37 rpm decreased the oil recovery by 0.2%/rpm, increased the throughput by 3.3 kg/rpm from 70 to 130 kg/h, and reduced the press load from 91 to 67%.

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## 1. Introduction

*Lesquerella fendleri* is an oilseed crop belonging to the Brassicaceae family that is native to the desert of the southwestern United States. The interest in this crop is due to the high level

of hydroxy fatty acids (HFA) in the oil. Lesquerella oil contains 54–60% lesquerolic (14-hydroxy-cis-11-eicosenoic) and 3–5% auricolic (14-hydroxy-11,17-eicosadienoic) acids (Hayes et al., 1995). HFA is used in a variety of industrial applications such as lubricants, corrosion inhibitors, engineering plastics,

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plasticizers, emulsifiers, and coatings. The current main source of HFA is castor oil which contains 90% ricinoleic (12-hydroxy-9-octadecanoic) acid.

The seed is about 1 mm in diameter and weighs about 0.63 g/1000 seeds. The seed contains 28% oil, 23 % protein, and 15% gums (Miller et al., 1962; Abbott, 1997). Glucoiberin (3(methylsulfinyl)propylglucosinolate) is the principal glucosinolate in lesquerella. The seed contains 65 mg glucoiberin/g of defatted meal (Vaughn and Berhow, 2005). The glucosinolates can be hydrolyzed enzymatically by thioglucosidase (TGSase) or thermally during seed processing into isothiocyanates, nitriles, thiocyanates, and other undesirable sulfur-containing compounds and may end up in the oil during extraction. The sulfur compounds in the oil impart objectionable taste, odor, and can poison catalysts during hydrogenation. Inactivation of TGSase is an important consideration in preparing the glucosinolates-containing seed for oil extraction.

Dry extrusion of coarsely ground soybeans followed by mechanical expelling of oil was proposed by Nelson et al. (1987). Extrusion offered a high temperature–short time cooking process in contrast to the long holding time and heating in conventional seed cookers. Extrusion increased the throughput of the expeller and the soybean oil produced had an oxidative stability comparable to refined deodorized oil. For small seeds like lesquerella, extrusion eliminates the flaking step because seed grinding and cooking are performed simultaneously.

Oil extraction from lesquerella seed by expelling, prepress-solvent extraction, full solvent extraction, and extrusion followed by full solvent extraction had been conducted but only the results of the latter had been published (Carlson et al., 1990; Lesquerella Task Force, 1991). This present study investigated the use of extrusion cooking as a method of preparing lesquerella seeds for full press oil extraction and how it affects the quality of the crude oil.

## 2. Materials and methods

### 2.1. Materials

Lesquerella seeds, harvested in 2005, were provided by Dr. David Dierig, USDA-ARS Arid Lands Agricultural Research Center, Maricopa, Arizona. The seeds were screened and aspirated to remove crop residue and other dockage. The seed was either used as is (6–7% moisture content (MC)) or adjusted to 12% MC as follows: predetermined amount of water was sprayed as the seeds (45 kg/batch) were loaded into a 200-L baffled stainless steel drum; the seeds were tumbled for 5 min and allowed to stand overnight; the seeds were tumbled again before unloading into 120-L container lined with 0.1 mm polyethylene bags and allowed to equilibrate for at least 24 h.

### 2.2. Seed cooking

#### 2.2.1. Extrusion cooking

The seeds were extruded using a Instapro Model 600 Jr (Instapro International, Des Moines, IA). The extruder was driven by a 37.3-kW (50 hp) motor with a variable frequency drive. The barrel was made up of an inlet and 3 compression

chambers held together by clamps (Fig. 1a). The die opening was 15.8 mm in diameter. The screw had 4 single-flighted screws (93.6 mm diameter, 126.7 mm long, and 32 mm pitch) separated by 82 mm steam locks (Fig. 1b). The steam from the extrudate was vented out through a 10-cm flexible tubing connected to a deflector/hood at the discharge end. The residence time in the extruder was varied by employing different screw speeds and feed rates. The extruder was allowed to run until the barrel temperature had stabilized (about 15–20 min), indicating that a steady state operation was reached. Extrusion rate was determined by collecting extrudate (extrusion-cooked seed) for 20 min. The amount of seed in the extruder barrel (holdup) was also determined after each run. The residence time was estimated by dividing the weight of the seed in the extruder barrel by the extrusion rate. The extrusion-cooked seed (ECS) temperature was recorded and its MC, protein solubility, and TGSase activity were analyzed.

#### 2.2.2. Cooking using a seed cooker

Cooked whole seed (CWS) were also prepared for screw pressing. CWS was cooked using a three-deck seed cooker (Laboratory Seed Cooker/Conditioner Model 324, French Oil Mill Machinery Company, Piqua, OH). Each deck can hold up to 0.08 m<sup>3</sup> (59 cm in diameter × 29.2 cm high) of material. The cooker was preheated using 0.34 MPa steam for about an hour before seed cooking. The sweeper arm on each deck (mounted to a common vertical shaft) guided the seed to the chute leading to the next deck below. To insure uniform cooking and drying, the seed in the cooker was recirculated by running the discharge screw and returning the seed to the top of the cooker. The seed was heated to 82 °C and cooked for another 20 min. The seed temperature was monitored and controlled to not exceed 110 °C thereby preventing overcooking or scorching. The cooked seed was unloaded into covered 120-L polyethylene containers and immediately screw pressed. The crude oils were analyzed as described above.

### 2.3. Screw pressing

The ECS and CWS were screw pressed immediately using a heavy duty laboratory screw press (Model L250, French Oil Mill Machinery Company, Piqua, OH). The detail of this expeller is available elsewhere (Evangelista and Cermak, 2007). The main screw was driven by a 14.9-kW (20 hp) motor equipped with a variable frequency drive. The cored main barrel (or cage) had a diameter of 8.9 cm. The barrel's length/diameter (L/D) ratio was 8.3. The cage had four drainage sections (14.0 cm/section). The screen bars in each section (feed to discharge) were spaced using 0.254-, 0.254-, 0.127-, and 0.076-mm shims. The screw consisted of alternating worms and collars (Fig. 2) for a severe shaft configuration. The compression ratio for this arrangement was 4. The main shaft rotational speed was 26 rpm, which was at the middle of the 20–30 rpm recommended by the manufacturer for full pressing. The cage was preheated to 82 °C using tempered water. A cone gap of 4.1 cm, measured between cone bracket and cone mounting plate, was used.

ECS and CWS were introduced slowly to the expeller, while monitoring the press load so as not to exceed 100%. The feed rate was controlled by a variable-speed screw conveyor. As soon as the feedworm was fully covered, water to cool the

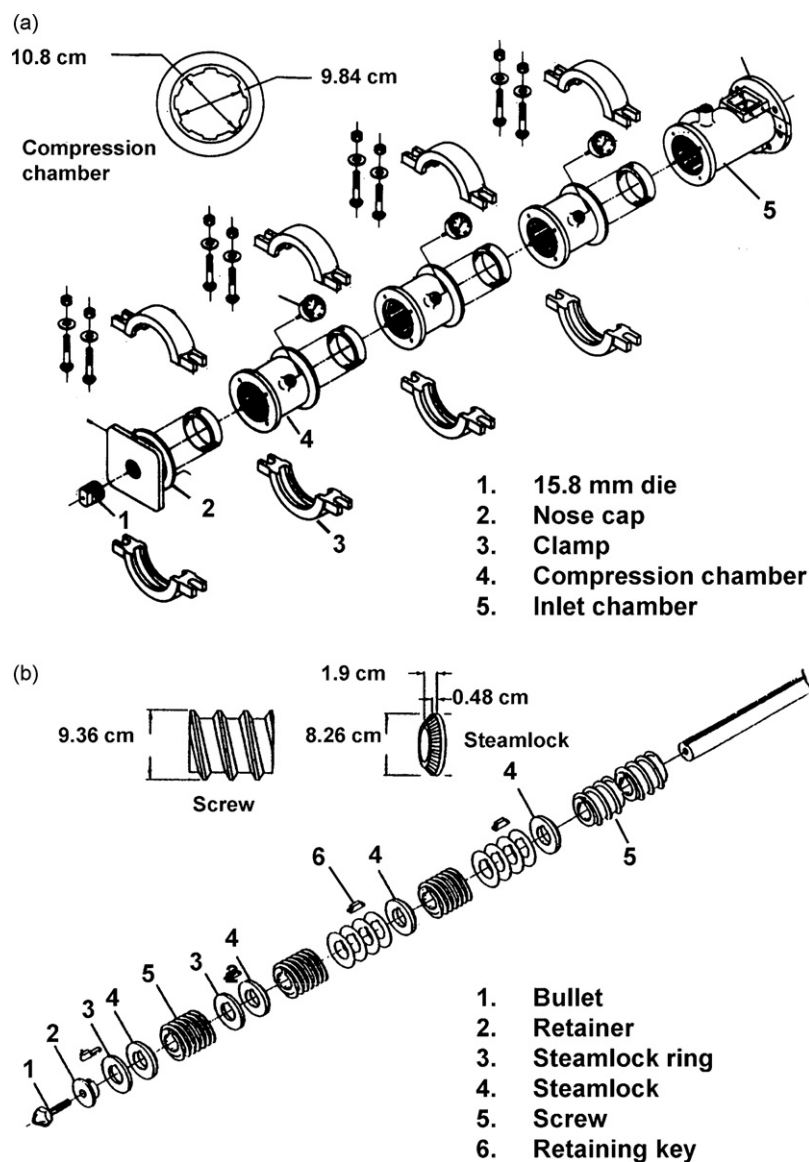


Fig. 1 – Instapro Model 600 Jr barrel (a) and screw (b) assemblies.

shaft was started. The shaft cooling outlet water temperature was maintained at about 60 °C. The press load reading was allowed to stabilize (about 10 min) before sampling. The oil was analyzed for solids (foots), free fatty acid (FFA), phosphorus, calcium, magnesium, and sulfur contents. Uncooked whole seed (UWS) was also screw pressed as control. The ECS was also pressed at different screw speeds and the press load, press rate, residual oil in the cake, and oil recovery were determined.

#### 2.4. Analytical methods

Moisture of the seed and press cake samples were obtained by following AOCS official method Ba 2a-38 (AOCS, 1997). The oil contents of the seed and press cake were determined using a pulsed NMR spectrometer (The Minispec, Bruker Optics Inc., Billerica, MA). Whole lesquerella seeds with known oil content were used to calibrate the spectrometer. The solids in

the crude oil were quantified using AOCS official method Ca 3a-46. The FFA (expressed as oleic) in the crude oil was determined using a Methrohm 702 SM Titrino (Methrohm, Ltd., Herisau, Switzerland) following AOCS official method Ca 5a-40. Phosphorus (for total phosphatide content) was determined by inductive coupled plasma optical emission spectroscopy (ICP-OES) (Perkin Elmer Optima 4300DV, Perkin Elmer, Wellesley, MA) by following AOCS official method Ca 20-99. Calcium and magnesium, a measure of nonhydratable phosphatides content (Hvolby, 1971), and sulfur in the crude oil were also analyzed by ICP-OES according to AOCS official method Ca 17-01.

For protein solubility, UWS, CWS, and ECS were ground in a coffee grinder and then defatted using hexane. Protein was extracted from 0.4 g of defatted sample with 20 mL sodium bicarbonate–carbonate buffer (pH 10). Lesquerella proteins were found to be most soluble at around pH 10 (Wu and Hojilla-Evangelista, 2005). Nitrogen content in the extract

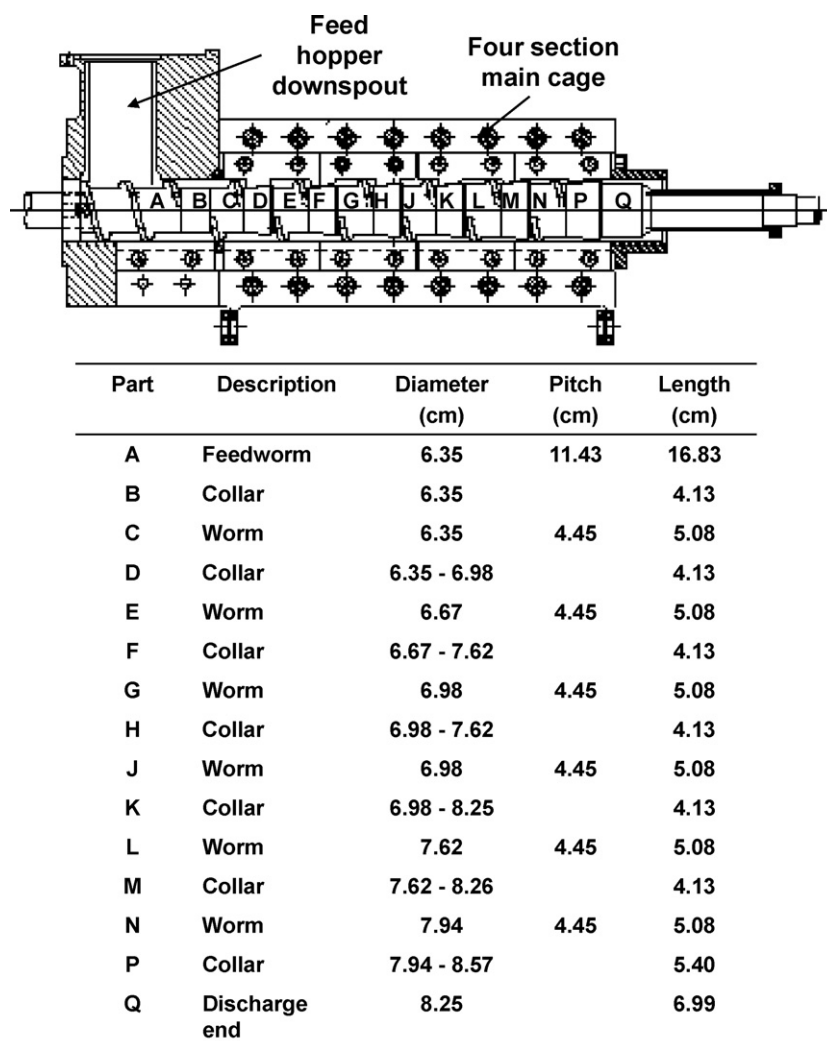


Fig. 2 – Screw configuration for French Model L-250 heavy duty laboratory screw press.

was analyzed by the Kjeldahl method and crude protein was calculated ( $\%N \times 6.25$ ). TGSase activity was determined qualitatively using glucose test strips (Clinistix® Reagent Strips, Bayer Corporation, Elkhart, IN) similar to what was used by Carlson et al. (1990). A slurry of 0.1 g ground seed in 1 mL distilled water was allowed to stand for 15 min before testing.

Statistical analyses were performed using PROC GLM in SAS v9.1 for PC (SAS Institute, Inc., 2006). ANOVA was conducted and the significant differences among treatments (duplicate replications) were determined using Duncan's Multiple Range test ( $P < 0.05$ ).

### 3. Results and discussion

Preliminary runs on the extruder were performed with a single-flighted screw followed by three double-flighted screws. This configuration, used successfully for soybeans, resulted in a backflow of oily cooked seed in the extruder barrel. This could be due to the higher oil content in lesquerella seed (28%) than in soybeans (21%). After several trials, a shaft configuration with four single-flighted screws and 15.8 mm die

was found suitable for extruding lesquerella seed in the range of screw speeds, feed rates, and seed moistures used in this study.

The different screw speed and feed rate combinations employed resulted in extrusion rates ranging from 62 to 296 kg/h (Table 1). The estimated residence times in the extruder varied from 22 to 110 s. Depending on residence time and starting seed moisture, ECS temperatures from 88 to 143 °C were achieved. The temperature of the ECS increased with increasing residence time. ECS with 6% starting MC also had higher temperatures than the seed with 12% MC at residence time  $\leq 80$  s. The moisture in the seed can act as a lubricant (Reuber, 1992; Singh et al., 2002), thus the drier seed imparted higher friction resulting in more heat being generated inside the barrel. ECS with 6% starting MC dried to 4.3% at temperatures between 111 and 125 °C regardless of residence time. The MC of the ECS with 12% starting MC decreased between 7 and 9% depending on the extrudate temperature and residence time. Further drying is required to bring the MC of the extrudate down to the optimum level (3–5%) for screw pressing. Semi-fluid ECS was obtained when the extrudate temperature was around 125 °C or higher. The expelled oil was



**Table 1 – Preparation of lesquerella seed for oil extraction**

Starting seed moisture (%)	Screw speed (rpm)	Feeder dial scale (1–100)	Extrusion rate (kg/h)	Residence time (s)	Extrudate temperature (°C)	Extrudate moisture (%)	Protein solubility <sup>a</sup> (%)	TGSase activity (+/–)
6	490	35	296	22	111 ± 0	4.3 ± 0.0	45.3 ± 0.3 c	(–)
12	490	35	282	27	88 ± 0	9.2 ± 0.2	44.9 ± 0.4 cd	(–)
6	490	25	187	34	118 ± 1	4.3 ± 0.0	39.5 ± 0.0 f	(–)
12	490	25	176	41	108 ± 0	8.1 ± 0.0	42.2 ± 0.3 def	(–)
6	490	14	68	80	143 ± 0	2.3 ± 0.1	26.1 ± 0.2 g	(–)
12	490	14	62	81	123 ± 1	7.1 ± 0.1	28.4 ± 1.3 g	(–)
6	270	14	65	107	125 ± 5	4.3 ± 0.0	23.0 ± 0.4 h	(–)
12	270	14	62	110	125 ± 1	7.3 ± 0.4	17.6 ± 0.5 i	(–)
6	Uncooked whole seeds (UWS)						56.2 ± 1.1 a	(+)
12	UWS						57.6 ± 1.8 a	(+)
6	Cooked whole seeds (CWS)			50 min <sup>b</sup>	107 <sup>c</sup>	5.11 ± 0.13 <sup>d</sup>	53.9 ± 0.1 b	(–)
12	Cooked whole seeds			47 min <sup>b</sup>	95 <sup>c</sup>	7.53 ± 0.01 <sup>d</sup>	41.6 ± 0.6 ef	(–)

<sup>a</sup> Means within a column followed by different letters are significantly different ( $P < 0.05$ ).

<sup>b</sup> Total time to heat seed to 82 °C + 20 min.

<sup>c</sup> Seed temperature at the end of cooking.

<sup>d</sup> Seed MC after cooking.

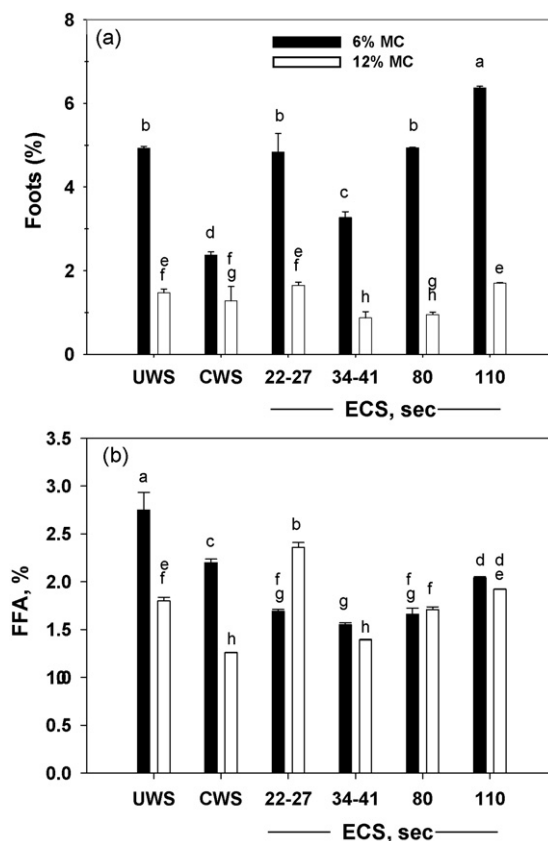
absorbed quickly by the ECS, which ended looking like loose ground seed. At temperatures  $<125^{\circ}\text{C}$ , the ECS was mostly in the form of short rods or pellets.

Protein solubility, an indicator of protein denaturation, was determined to assess the degree of cooking as a result of heat treatment. The protein solubility of UWS was 57%, which is consistent with what was reported by Wu and Hojilla-Evangelista (2005). CWS with starting MC of 6 and 12% had 5 and 20% drop in protein solubility, respectively (Table 1). For the ECS, the solubility of the protein decreased as the residence time and extrudate temperature increased. Significant difference in protein solubility between 6 and 12% MC seeds occurred only at residence time of about 110 s where a drop of 60 and 68%, respectively, were observed. Seed with 6 and 12% MC extruded at a residence time of 34 and 41 s (extrudate temperatures of 118 and  $108^{\circ}\text{C}$ ), respectively, provided the same degree of cooking (same protein solubility) as that of the 12% MC CWS. All ECS and CWS tested negative for TGSase activity. Carlson et al. (1990) reported that TGSase in flaked lesquerella seed (10% MC) was inactivated when extruded at  $77^{\circ}\text{C}$  and 34 s residence time.

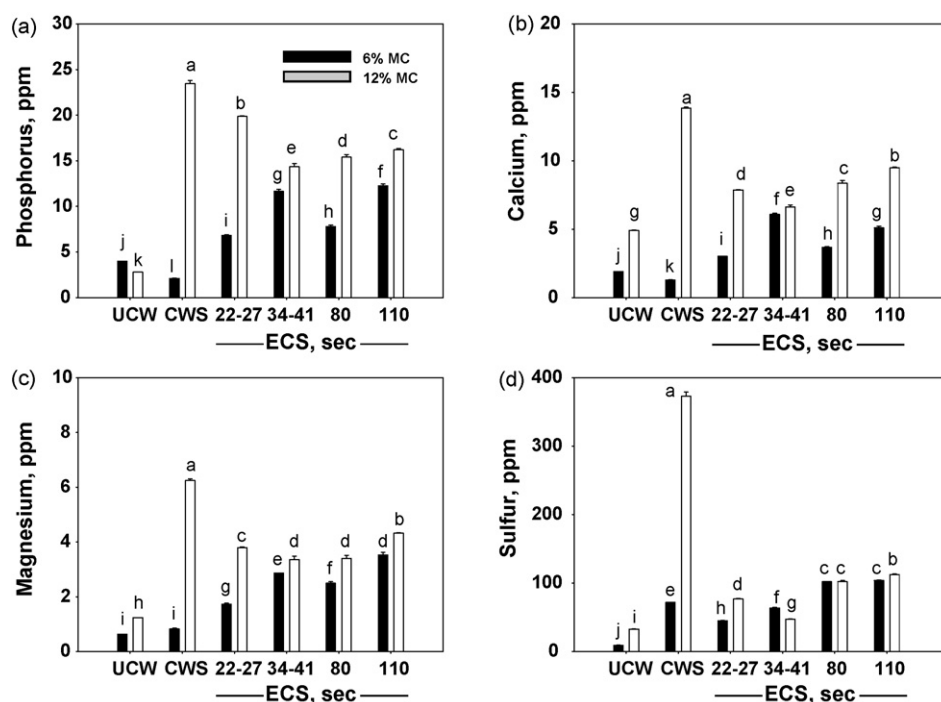
Expelling lower moisture UWS, CWS, and ECS (6% starting MC) produced considerably higher foots in the crude oil than those of their corresponding 12% MC seed (Fig. 3a). This inverse relationship between seed MC and foots in the oil has been reported for crambe and cuphea (Singh et al., 2002; Evangelista and Cermak, 2007). In a well run rapeseed prepress plant, foots content in the expelled oil is 3–5% (Ward, 1984). Crude oils from CWS and ECS tend to have lower FFA contents than the oils from UWS (Fig. 3b). The lowest FFA (1.25%) was from CWS with 12% starting MC. The FFA content (1.6–2%) in the crude oil from the ECS with 6% MC did not appear to be affected by residence time and temperature. On the other hand, FFA content in the crude oil from the 12% MC ECS varied from 1.3 to 2.4%.

Phosphorus (P) is a measure of total phosphatides in the oil. Calcium (Ca) and magnesium (Mg) are associated with the nonhydratable phosphatides (NHP). The phosphatides are removed in the degumming step during the oil refining

process. The NHPs are removed by treating the oil with phosphoric or citric acid to chelate Ca and Mg in NHP and render it hydratable. As shown in Fig. 4a, cooking and higher MC significantly increased the P content in the crude oil. P contents in crude oil from CWS and ECS with 12% starting MC were also higher than those obtained from seeds with 6% starting



**Fig. 3 – Foods (a) and FFA (b) contents of crude oil from uncooked and cooked lesquerella seeds. Bars with different letters are significantly different ( $P < 0.05$ ).**



**Fig. 4 – Phosphorus (a), calcium (b), magnesium (c), and sulfur (d) contents of crude oil from uncooked and cooked lesquerella seeds. Bars with different letters are significantly different ( $P < 0.05$ ).**

MC. Crude oil from seed with 12% MC CWS had the highest P content at 23 ppm which was 8x higher than that of the UWS. Ca and Mg in the crude oil also increased with increase in MC and cooking (Figs. 4b and c). However, the increase was not due to conversion of hydratable phosphatides to NHP because the ratios of Ca and Mg to their corresponding P in CWS and ECS were about the same. Phospholipase D catalyzes the conversion of hydratable phosphatide to NHP (List et al., 1992). It is very likely that phospholipase D was also inactivated during cooking.

The amount of P in the crude lesquerella oil was lower compared to other oils (Table 2). P, Ca, and Mg values were comparable to totally degummed rapeseed oil. Therefore, degumming may be unnecessary in refining crude lesquerella oil.

As with the other seeds containing glucosinolates, preparation of lesquerella seed for oil extraction is a major concern. TGSase catalyzes the hydrolysis of glucosinolates into isothiocyanates, nitriles, thiocyanates, and other sulfur-containing compounds. Thermal degradation of glucosinolates also produces isothiocyanates and nitriles (MacLeod et al., 1981). In rapeseed oil, the isothiocyanates can poison catalyst used during hydrogenation. Inactivation of TGSase is influenced largely by temperature, heating time, and seed moisture. Seed cooking must be carried out in such a way that TGSase is inactivated but not severe enough to cause thermal decomposition of glucosinolates.

As shown in Fig. 4d, the oil with the lowest amount sulfur was from 6% MC UWS. By increasing the MC of UWS to 12%, the sulfur content in the oil increased from 9 to 33 ppm.

**Table 2 – Phosphorus, calcium, and magnesium contents of selected crude oil from pressed seeds**

Oils	Phosphorus (ppm)	Calcium (ppm)	Magnesium (ppm)
Lesquerella oil			
Crude oil	<25	<15	<5
Rapeseed oil <sup>a</sup>			
Crude oil	530–630 <sup>b</sup>	–	–
Water-degummed oil	142–152	107–188	18–34
Total degummed oil	21–70	15–23	2–9
Soybean oil <sup>a</sup>			
Crude oil	630–940 <sup>c</sup>	70–200	50–150
Water-degummed oil	90–250	50–120	20–100

<sup>a</sup> Serger and van de Sande (1989).

<sup>b</sup> Expelled oil (Thomas, 1982).

<sup>c</sup> Converted from % phosphatide.

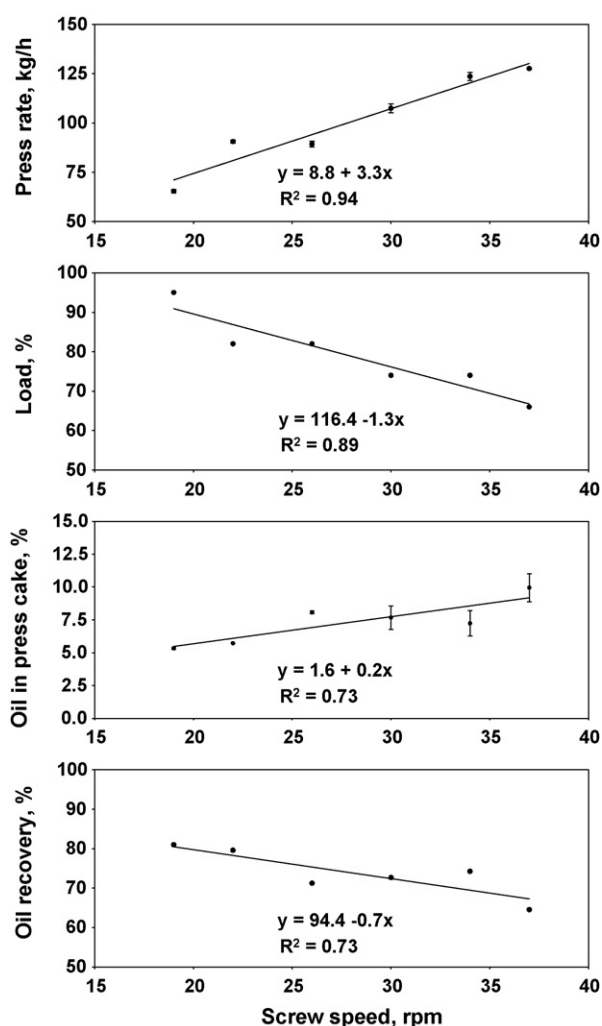


Fig. 5 – Expelling extruded 6% MC lesquerella seed at different screw speed: effect on press rate (a), press load (b), residual oil in press cake (c), and oil recovery (d).

The study by Daun and Hougen (1976) showed that, even without heat treatment, merely increasing the MC of rapeseed resulted in higher sulfur content in the oil. Cooking the seed increased the sulfur content in the oil. The crude oil from 12% MC CWS had 374 ppm of sulfur, the highest among cooked seeds. High MC in the seed and the long heating time in the seed cooker favored the thermal degradation of glucosinolates. Seeds extruded at residence times  $\leq 40$  s had significantly lower sulfur (45–77 ppm) in their oils compared with the oils from seeds extruded at higher residence times. Starting MC of the seed had very significant effect on the sulfur level in the oils from CWS but not on ECS. Depending on seed variety, crude oil from expelled rapeseed contains 19–25 ppm of sulfur (Daun and Hougen, 1976). This difference in sulfur level is due to the higher glucosinolate content of lesquerella seeds (65 mg/g defatted meal) than rapeseed (21–29 mg/g defatted meal).

Lesquerella seed with 6% starting MC was extruded at 490 rpm and 35 scale on the feeder dial (for 22 s residence time) and expelled at different screw press speeds. The screw

Table 3 – Press performance at 26 rpm screw speed when pressing cooked whole seed and extruded seed

	Cooked whole seed	Extruded seed
Press rate (kg/h)	$76.0 \pm 2.0$	$89.3 \pm 3$
Press load (%)	$76 \pm 2$	$80 \pm 2$
Oil in press cake (%)	$5.2 \pm 0.0$	$7.9 \pm 0.3$
Oil yield (%)	$81.4 \pm 0.1$	$71.8 \pm 1.1$

speed ranged from 19 to 37 rpm, which covered speeds suitable for full pressing and prepressing operations. As shown in Fig. 5a, increasing the screw speed increased the press rate by 3.3 kg/rpm from 70 to 130 kg/h. The load to the press (expressed as percentage of the 24.8 A full-load current drawn by the motor driving the screw) decreased from 91 to 67% as the screw speed increased (Fig. 5b). However, the residual oil in the cake also increased by 0.2%/rpm (Fig. 5c). This increase in residual oil decreased the oil recovery by 0.7%/rpm (Fig. 5d). The decrease in press load was indicative of the reduced friction between the screw and the seed as the residual oil increased. Increasing the screw speed reduced the residence time of the seed inside the barrel, hence the time for the oil to drain from the pressed seed also decreased. Vadke and Sosulski (1988) reported that the pressure in the barrel decreased with increasing screw speed when pressing canola. Their study also showed that the residual oil in the cake increased as the screw speed increased.

The press rate of ECS was 18% higher than that of the 6% MC CWS when screw pressed at 26 rpm (Table 3). However, the CWS had a 34% higher oil recovery than the ECS. To obtain the same oil recovery as the CWS, the ECS had to be pressed at a lower speed (19 rpm), thus reducing the press rate. For ECS, a press with higher L/D and compression ratio may be needed to attain lower residual oil at a higher press rate.

#### 4. Conclusions

Extrusion cooking is a suitable process for preparing lesquerella seeds for oil expelling. Inactivation of TGSase can be accomplished even at 6% seed MC and 22 s of residence time. Extrusion and expelling of lesquerella seeds with 6% MC produced oil with lower sulfur compared with the oil from seeds cooked in the seed cooker. The phosphatides content in the oil from extrusion and expelled seed were higher but still comparable with super degummed oils. However, extrusion cooked seeds will require an expeller with higher L/D ratio and/or higher screw compression ratio than what was used in this study to improve oil recovery and maintain high throughput. A tighter lining bar spacing also reduces the levels of foots present in the oil. The effect of extrusion and expelling on the quality of press cake and in seed gum recovery has to be evaluated to better assess the merits of this process.

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